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PART II

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DEVELOPMENT OF A GAS TURBINE ENGINE OIL FOR BULK OIL TEMPERATURES OF -40 TO 465° F
PART II

MONSANTO RESEARCH CORPORATION

DECEMBER 1975

TECHNICAL REPORT AFML-TR-247, PART II
FINAL REPORT FOR PERIOD OCTOBER 1974 - SEPTEMBER 1975

AIR FORCE MATERIALS LABORATORY
AIR FORCE WRIGHT AERONAUTICAL LABORATORIES (AFSC)
Air Force Systems Command
Wright-Patterson Air Force Base, Ohio 45433

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This technical report has been reviewed and is approved for publication.

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- MIL-L-27502 bearing tests
- Ryder gear tests
- Storage life tests
- Magnesium deactivators
- Oxidation-corrosion tests

20. ABSTRACT (Continue on reverse side if necessary and identify by block number):

This contract involved advanced characterization of three MIL-L-27502 lubricants. These were ester blends designated MCS 1710, MCS 1709 and Fluid 12. Oxidation-corrosion and storage life tests were run on all fluids. MIL-L-27502 bearing tests were run on MCS 1710 and MCS 1709; each gave very good results. Moreover, both of these blends have satisfactory Ryder loads.
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20. ABSTRACT (continued)

Two hundred gallons of MCS 1710 plus a metal deactivator were blended and sent to AFML for further evaluation. The deactivator was added to reduce magnesium corrosion of MCS 1710.

PREFACE

The research work described in this report was performed by Monsanto Research Corporation under Modification No. P00003 to contract F33615-73-C-5079. That contract was initiated under Project No. 7343, "Aerospace Lubricants", Task No. 734303 "Fluid Lubricant Materials", with the Air Force Materials Laboratory, Wright-Patterson Air Force Base, Ohio. Mr. George J. Morris (MBT) was the laboratory project monitor for both the initial contract and the modification. Unless stated otherwise, the work was done by Monsanto Research Corporation at the Monsanto Company Research Center, St. Louis, Missouri; it began on 1 October 1974 and ended 22 September 1975.

Mr. John D. Hinchen assisted this research with several statistical analyses of the oxidation-corrosion tests. This included the regression analysis of magnesium corrosion. The oxidation tests were run by Mr. William C. Warren. This report was submitted for approval on December 1975.

Part I of this technical report has the same title and report number as this report. It was issued in April 1975 and describes the studies done during the initial contract.

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LIST OF ABBREVIATIONS

AFML	Air Force Materials Laboratory
AMS	Aeronautical Material Specifications
ASTM	American Society for Testing and Materials
BrI	Bronze I (4616 bronze)
BrII	Bronze II (Mueller 600, alloy No. 674)
MCS	Monsanto Company sample
MRC	Monsanto Research Corporation
NC	No change
σ	Standard deviation
TAN	Total acid number
WSP	Waspalloy

I. INTRODUCTION

This contract was a continuation of contract F33615-73-C-5079. The objective of both programs was to produce a new gas turbine engine oil for use at bulk temperatures of -40°F to +465°F (-40°C to +240°C). Currently available engine oils and their bulk temperature limits include:

Specification MIL-L-7808	-65°F to +325°F (-54°F to +163°C)
Specification MIL-L-23699	-40°F to +350°F (-40°F to +177°C)

Recent studies by the Air Force Aeropropulsion Laboratory show that new or improved engine oils having still higher bulk temperature capabilities are needed for advanced engine designs; hence the goal of a -40°F to +465°F fluid - Specification MIL-L-27502.

In the initial contract (Ref. 1) a screening program evaluated various reformulations of a reference MIL-L-27502 oil. The main screening tests were oxidation-corrosion tests (modified Federal Test Method 5307), storage life tests, and a specification of a 1 centistoke viscosity minimum at 500°F. This produced two fluids with properties superior to those of the reference oil. These were designated MCS 1710 and MCS 1709. After their selection, a thorough delineation of these blends followed; this included 96-hr oxidation-corrosion tests and 15 physical/chemical property tests based on MIL-L-27502 guidelines.

The present contract continued characterization of these fluids. The tests employed were:

Oxidation-corrosion tests (Federal Test Method 5307)

Ryder Gear Tests

MIL-L-27502 Bearing tests

Storage tests

In addition, more oxidation-corrosion tests were run on the reference oil used in the previous contract. Accumulated storage data showed that this ester (designated Fluid 12) had outstanding storage stability. Thus it was considered not only a reference blend but one of the candidate fluids for use at 465°F.

II. SUMMARY

A. MCS 1710

This ester had excellent stability in high temperature (464°F) oxidation-corrosion tests. It gave a very good MIL-L-27502 bearing test, the outstanding features of which were low filter deposits and a low deposit rating. It has a satisfactory Ryder gear load and good storage life. At a fluid temperature of 428°F, MCS 1710 causes some magnesium corrosion in a macro oxidation-corrosion test, but this corrosion does not occur at 418°F. Metal deactivators greatly reduce the attack on magnesium at 428°F.

B. MCS 1709

MCS 1709 gave excellent results in a MIL-L-27502 bearing test showing low deposits, low acid number, low viscosity increase, and little metal corrosion. It showed good stability in 464°F oxidation-corrosion tests; some magnesium corrosion occurred at 428°F but none at 418°F. It has a satisfactory Ryder gear load with a storage life slightly inferior to that of MCS 1710.

C. Fluid 12

This blend* has excellent storage life. It is marginal with respect to magnesium corrosion at 428°F. Its viscosity increase during oxidation-corrosion tests is anomalous.

A 200-gal blend of MCS 1710 containing a metal deactivator was prepared and forwarded to the Air Force Materials Laboratory for further evaluation. Magnesium corrosion by this 200-gal lot was somewhat higher than for laboratory samples (-1.0 mg/cm^2 vs. -0.1 mg/cm^2 after 72 hr).

The conclusions and recommendations based on this work are given in Section III. Subsequent sections describe:

- oxidation-corrosion tests on the candidate fluids (Section IV)
- bearing tests (Section V)
- Ryder gear tests (Section VI)
- two approaches to eliminating the magnesium corrosion of MCS 1710 (Section VII)

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*L-71 ester plus 5% L-31 ester plus a standard additive package.

- oxidation-corrosion tests on the 200-gal sample (Section VIII)
- storage life tests (Section IX)

Section IX also includes storage data carried over from the initial contract. As mentioned previously, Fluid 12 exhibits outstanding storage life. However, one of the base stocks for this blend, L-71 ester, is no longer commercially available. A substitute product, L-100, is available. Storage data on selected blends in this base stock are reported in Section IX.

III. CONCLUSIONS AND RECOMMENDATIONS

A. CONCLUSIONS

(1) MCS 1710 and MCS 1709 are outstanding MIL-L-27502 oils. Their oxidative stability is shown in oxidation-corrosion tests at 428°F and 464°F and in bearing deposition tests. The stability of MCS 1710 in the 464°F oxidation-corrosion test is particularly impressive.

(2) Critical fluid temperatures exist above which MCS 1710 and MCS 1709 will corrode magnesium in a 72-hr macro oxidation-corrosion test. The critical temperature is around 420°F for MCS 1710 and around 430°F for MCS 1709. Several metal deactivators greatly reduce the magnesium corrosion of MCS 1710. At least one of these can be put into MCS 1710 without harmful side effects.

(3) Fluid 12* and L-100, a new, commercially available ester basestock, have superior storage compatibility with the additive packages under study.

B. RECOMMENDATIONS

(1) Further developmental work on MCS 1710 and/or MCS 1709 should cover engine tests, toxicological screening, and reclamation studies.

(2) Blends of selected additives in L-100 should be further characterized by oxidation-corrosion tests, storage tests, Ryder gear, and MIL-L-27502 bearing tests.

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*L-71 ester plus 5% L-31 ester

IV. OXIDATION-CORROSION TESTS ON CANDIDATE FLUIDS

The oxidation-corrosion tests of the previous contract used a modified Federal Test Method Standard 791b, Method 5307 procedure. After completion of that contract the Air Force Materials Laboratory (AFML) reported some different results in their oxidation-corrosion tests from those of the modified procedure. In particular the AFML tests showed that MCS 1710 corroded magnesium in micro and some macro tests at 428°F after 72 hours. In MRC's oxidation-corrosion tests this fluid passed the MIL-L-27502 magnesium corrosion specification of ± 0.2 mg/cm^2 after 96 hours. AFML observed no metal corrosion after 48 hours at 464°F by MCS 1710 (micro oxidation-corrosion rig); after 72 hours this oil had a bronze loss of -16.0 mg/cm^2 and a mild steel loss of -0.26 mg/cm^2. There was negligible corrosion of bronze and mild steel after 96 hours in an MRC test.

Because of these discrepancies and because all these oxidation tests were run using different procedures, it was decided to rerun all macro tests on the candidate fluids according to Federal Test Method 5307. The results confirm magnesium corrosion by MCS 1710 at 428°F, but show no bronze or steel corrosion at 464°F. Indeed, the high temperature (464°F) stability of MCS 1710 in both the 5307 and the modified 5307 tests is an outstanding property of this oil. A discussion and comparison of the two test methods is provided in Appendix A.

A. 428°F (72 HOURS)

Data for four tests on MCS 1710, four tests on MCS 1709, and five tests on Fluid 12 are given in Table 1. The values given are from individual tubes of a given test, with each test utilizing two tubes of each fluid.

1. MCS 1710

No significant metal corrosion occurred except for magnesium which had an average weight loss of 13 mg/cm^2. This is in good agreement with some of the values obtained at AFML. Since MCS 1710 produced a magnesium corrosion of only -0.1 mg/cm^2 after 96 hours at a fluid temperature of 418°F*, it can be speculated that there is a critical temperature around 420°F above which the fluid begins to attack the magnesium metal. The percent viscosity increase for MCS 1710 (14.7%) is not substantially different from that for MCS 1709 (11.2%).

*The fluid temperature of the modified procedure (see Appendix A).

TABLE 1

OXIDATION-CORROSION TESTS AT 428°F (5307 METHOD)
(72 HOURS)

MCS 1710 Run No.	% Viscosity Increase		TAN		Mg Change (mg/cm^2)	
1	12.6	14.1	0.5	0.5		*
2	15.4	5.1	9.8	6.5	- 8.7	-11.9
3	18.2	18.6	4.1	4.8	-13.7	-14.8
4	16.9	16.9	2.0	2.8	-13.0	-15.7
	av = 14.7		av = 3.9		av = -13.0 σ = 2.5	
MCS 1709						
1	14.1	13.3	0.3	0.2		*
2	10.2	9.7	2.4	2.2	- 6.4	- 5.5
3	11.2	11.5	1.4	1.6	- 0.1	- 0.3
4	9.9	9.9	2.2	1.5	- 3.1	- 2.6
	av = 11.2		av = 1.5		av = - 3.0 σ = 2.6	
Fluid 12						
1	15.9	13.3	0.4	0.5		*
2	1.7	6.9	0.6	4.5	- 1.9	- 6.0
3	8.7	10.1	1.3	1.5	NC	- 0.1
4	3.8	8.0	1.1	0.8	- 1.8	- 0.5
5	- 0.8	8.7	0.4	0.3	- 1.4	- 0.1
	av = 7.6		av = 1.4		av = - 1.5 σ = 2.0	

*Metal loss not obtained due to balance malfunction.

Figure 1 shows a plot of viscosity increases with respect to time for an oxidation-corrosion test on the candidate fluids. No sharp break-point occurs with any fluid through 72 hours.

2. MCS 1709

This fluid had an average magnesium corrosion of -3.0 mg/cm^2, with one test in three being within the MIL-L-27502 specification. AFML did not observe magnesium corrosion with this oil. The critical temperature, if it exists, is perhaps around 430°F, above which magnesium corrosion occurs. The viscosity increase vs. time plot is also shown in Figure 1. The weight loss for all other metals was within specifications.

3. Fluid 12

This ester resembles MCS 1709 in that the magnesium corrosion is sometimes within the MIL-L-27502 specification but usually is slightly above this specification. The average corrosion for five tests was -1.5 mg/cm^2. The viscosity increase shows a wide range with an occasional very low value.

B. 464°F (72 HOURS)

Data for the three candidate fluids are given in Table 2. The numbers represent individual tubes of a given oxidation-corrosion test; two tubes of each fluid were used per test.

1. MCS 1710

This oil gave low viscosity increases (27.8% overall) with no break-point as a function of time (see Figure 2) It produced no bronze corrosion. Micro oxidation-corrosion tests at AFML did show bronze corrosion for MCS 1710 after 72 hours but not after 48 hours.

2. MCS 1709

This blend gave a slightly higher viscosity increase than MCS 1710 (43% vs. 28%) with no break-point as a function of time (see Figure 2). Bronze corrosion was within specification.

3. Fluid 12

This oil gave no excessive bronze corrosion. The viscosity increase range was very large, going from -0.2 to +74.9%. This is partly explained by the plot of viscosity increase vs. time (Figure 2), which shows a sharp break around 60 hours. Slight changes in the position of the break would give large changes in the absolute value of the viscosity at 72 hours. However, this does not account for the tubes giving viscosity increases below 5%.

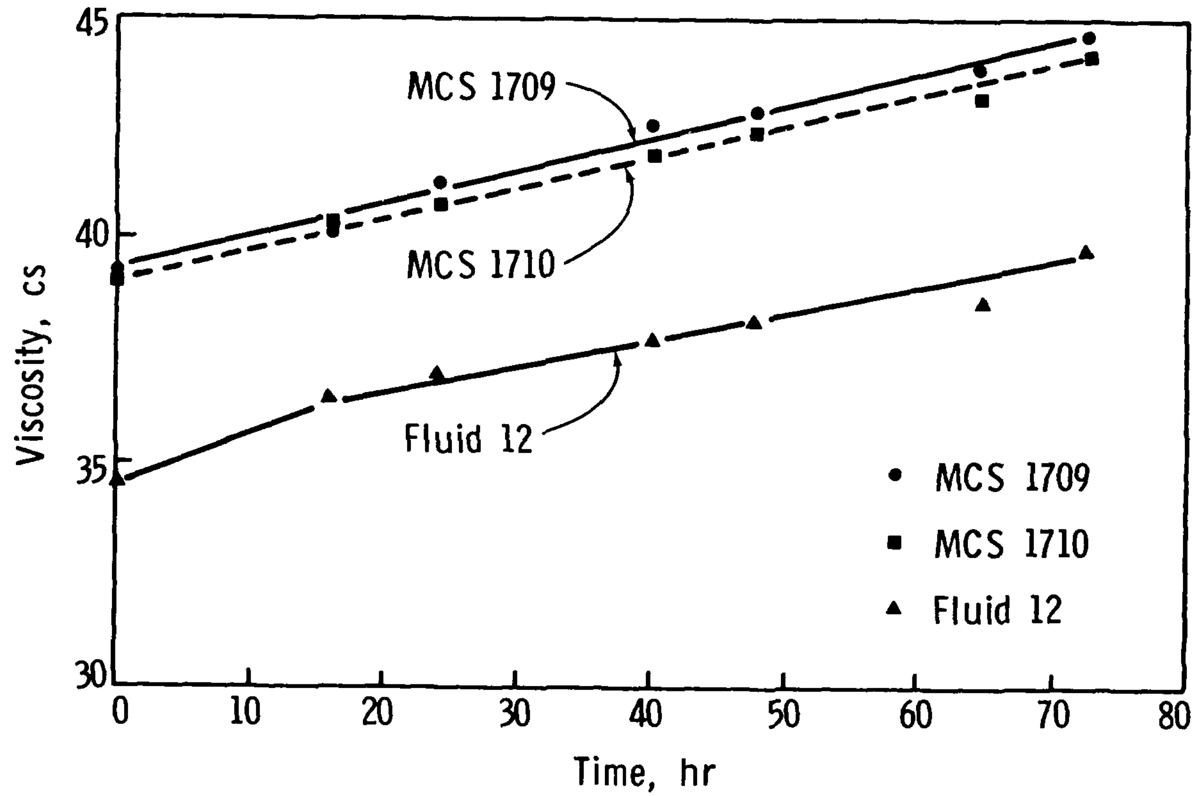

Figure 1. Viscosity vs Time During a 428°F Oxidation-Corrosion Test (Run #1)

TABLE 2

OXIDATION-CORROSION TESTS AT 464°F (5307 METHOD)
(72 HOURS)

MCS 1710 Run No.	% Viscosity Increase		TAN		Br^{II} Change (mg/cm^2)	
1	29.6	28.6	5.9	6.6	NC	+0.04
	23.2*	29.6*	5.9*	6.2*	+0.1	+0.1*
	av = 27.8		av = 6.2			
MCS 1709						
1	46.4	40.3	6.8	8.8	+0.0	-0.3
	av = 43.4		av = 7.8			
Fluid 12						
1	74.9	52.0	13.1	11.4	-0.1	-0.1
2 (bath 2)	26.3	2.3*	7.5*	0.9*	-0.1*	-0.2*
3 (bath 2)	54.8	- 0.2	12.0	0.9	-0.3	-0.3

*Air cooling

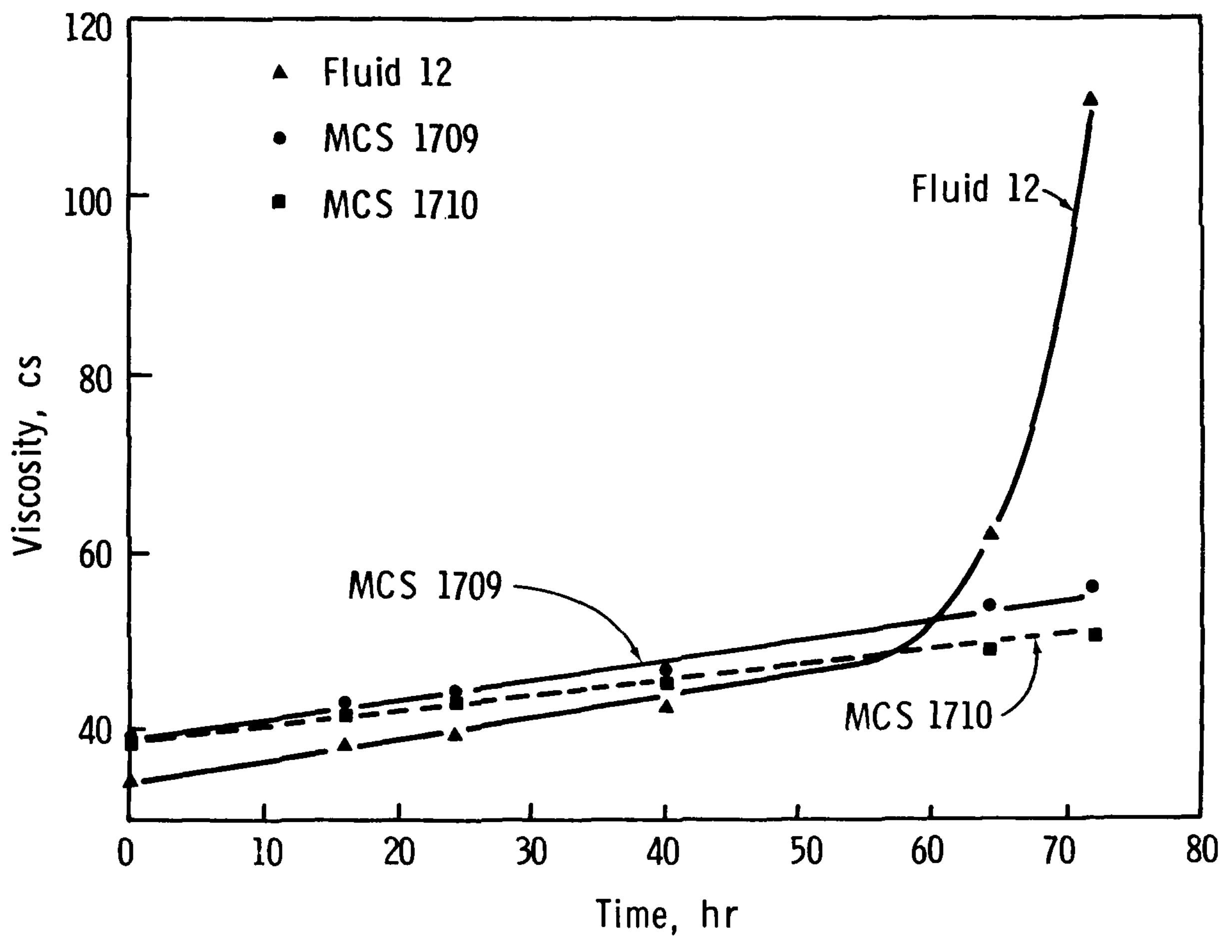

Figure 2. Viscosity vs Time During a 464°F Oxidation-Corrosion Test (Run #1)

V. ERDCO BEARING TESTS

These tests are run at medium to high temperatures to gauge the deposit formation and overall oxidative stability of an oil. The test method is defined in Appendix I of Technical Report AFAPL-TR-67-85 (July 1967). The bearing was an unshielded, 100 mm straight roller bearing, Rollway P/N RCS 120-560*. The tests were run by Alcor, Inc., of San Antonio, Texas.

A. MCS 1710

Two deposition tests were run on this blend. The conditions for one were those of specification MIL-L-27502, for the other those of specification MIL-L-7808.

1. MIL-L-27502 Test

This test is run for 48 hours at high temperatures:

Oil-in temperature	455°F
Bearing temperature	572°F
Bulk oil temperature	464°F

The test results are summarized in Table 3. The fluid passed all of the MIL-L-27502 specifications; in particular, it had a low deposit rating and very little filter sludge. This is emphasized in Table 4, which compares the bearing test results of three ester blends. MCS 1034 is the developmental oil which was optimized into MCS 1710 and MCS 1709 in the first contract.

Table 5 gives the demerit rating summary. The oil performance (viscosity and acid number change with time) is given in Table 6 and Figure 3.

2. MIL-L-7808 Test

This is a milder test run at the following conditions for 48 hours:

Oil-in temperature	340°F
Bearing temperature	400°F

*Rollway Bearing Co., Dallas, Texas

TABLE 3

MIL-L-27502 BEARING TEST RESULTS (MCS 1710)

	MCS 1710	Specification
Deposit Rating	48.5	80
Viscosity Increase (%)	64.8	100
Δ TAN	1.99	2.0
Filter Sludge (g)	0.335	2.5
(inlet screen)	0.143	
(outlet screen)	0.192	
Oil Consumption (ml)	1670	3600
Metal Wt. Change (mg/cm^2)		±0.2
Aluminum	-0.050	
Titanium	-0.004	
Silver	-0.024	
Steel	-0.016	
M-50 Steel	-0.040	
Bronze	-0.046	
Waspaloy	-0.024	

TABLE 4

COMPARISON OF MIL-L-27502 BEARING TESTS

	MCS 1709	MCS 1710	MCS 1034*	Specification
Deposit Rating	30.6	48.5	69	80
Viscosity Increase (%)	28.3	64.8	65	100
Δ TAN	0.96	1.99	1.44	2.0
Filter Sludge (g)	0.751	0.335	2.2	2.5
(inlet screen)	0.285	0.143		
(outlet screen)	0.466	0.192		
Oil Consumption (ml)	1950	1670		3600

*Developmental oil optimized into MCS 1710 and MCS 1709

TABLE 5

MIL-L-27502 BEARING TEST, MCS 1710
DEMERIT RATING SUMMARY

Item	Demerits	Factor	Rating
End Cover	3.0	1	3.0
Spacer and Nut	28.5	2	57.0
Heater - Front	32.4	3	97.2
Heater - Rear	23.5	3	70.2
Seal Plate	10.0	1	10.0
Bearing	10.7	5	53.5

OVERALL RATING: $\frac{290.9}{6} = 48.5$

TABLE 6

MIL-L-27502 BEARING TEST, MCS 1710
OIL PERFORMANCE

Hours	Viscosity @ 100°F (cs)	Viscosity Increase (%)	Total Acid No.
0:00	40.0		0.11
4:00	40.0	0.0	0.53
8:00	40.7	1.8	0.56
12:00	42.4	6.0	0.59
16:00	44.1	10.3	0.65
20:00	45.4	13.5	0.84
24:00	47.3	18.3	0.95
28:00	50.0	25.0	1.15
32:00	53.6	34.0	1.51
36:00	54.4	36.0	1.65
40:00	57.8	44.5	1.80
44:00	61.8	54.5	1.96
48:00	65.9	64.8	2.10

VISCOSITY INCREASE,	
48 hr. (cs)	25.9
48 hr. (%)	64.8
TAN INCREASE	1.99

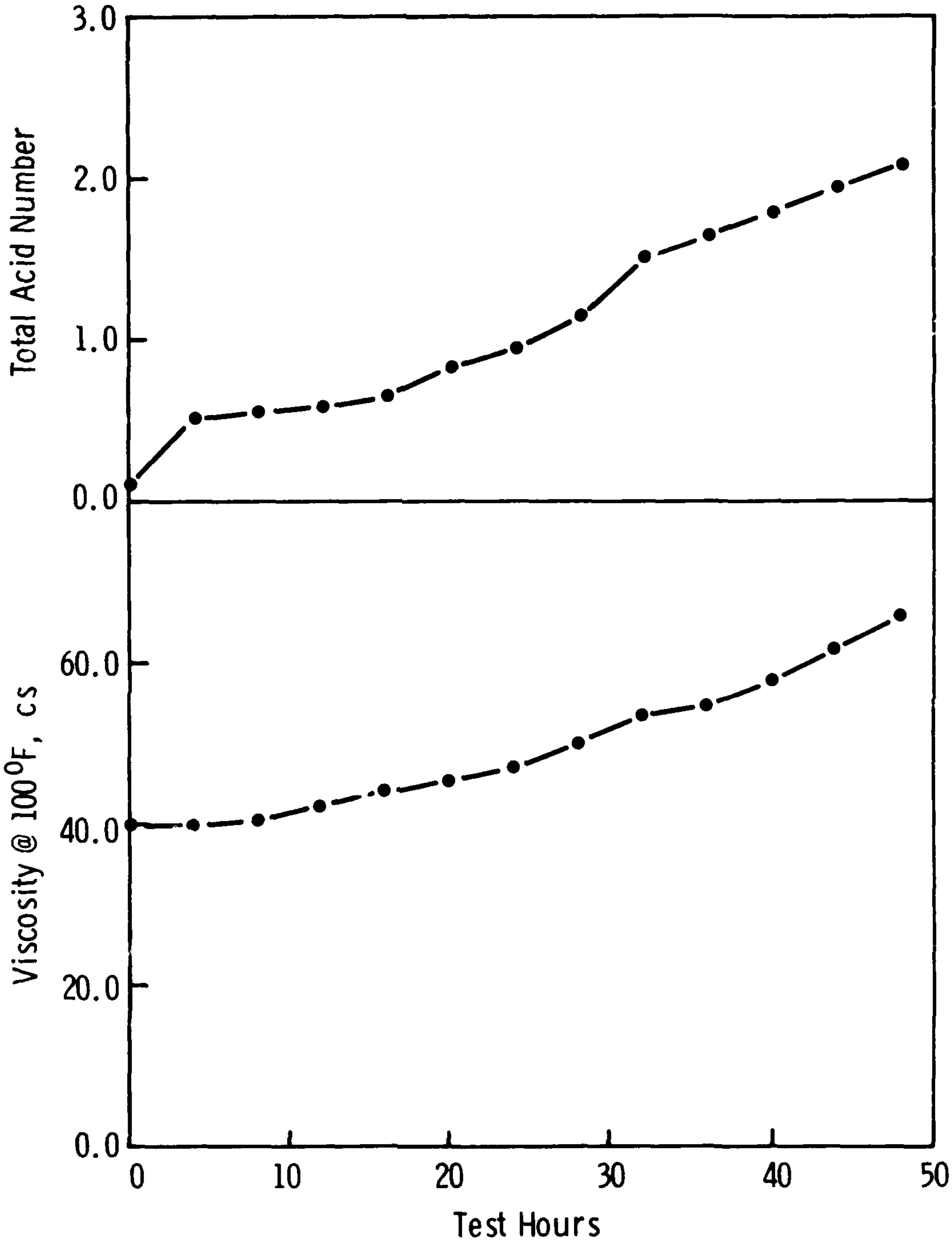

Figure 3. Plot of Viscosity and Total Acid Number vs Time for MCS 1710 (MIL-L-27502 Bearing Test)

Bulk oil temperature 350°F

The result using MCS 1710 (Table 7) was a very clean test.

B. MCS 1709 (MIL-L-27502 Test)

This fluid gave a low deposit rating, low viscosity increase, low acid number increase, low filter deposit, and little metal corrosion. Test results are given in Table 8, similar data on other esters are reported in Table 4.

Additional test information includes the deposit rating summary (Table 9) and the oil performance (viscosity and acid number change with time) (Table 10 and Figure 4).

TABLE 7

MIL-L-7808 BEARING RIG TEST RESULTS (MCS 1710)

Deposit Rating	27.6
Viscosity Increase, (%)	4.4
TAN Increase	0.59
Total Consumption, (cc)	455
Sludge	
Inlet Screen, (g)	0.159
Outlet Screen, (g)	0.223

TABLE 8

MIL-L-27502 BEARING TEST RESULTS (MCS 1709)

	MCS 1709	Specification
Deposit Rating	30.6	80
Viscosity Increase (%)	28.3	100
Δ TAN	0.96	2.0
Filter Sludge (g)	0.751	2.5
(inlet screen)	0.285	
(outlet screen)	0.466	
Oil Consumption (ml)	1950	3600
Metal Wt. Change, (mg/cm^2)		±0.2
Aluminum	+0.010	
Titanium	+0.014	
Silver	-0.079	
Steel	+0.020	
M-50 Steel	+0.063	
Bronze	+0.010	
Waspaloy	+0.032	

TABLE 9

MIL-L-27502 BEARING TEST, MCS 1709
DEMERIT RATING SUMMARY

Item	Demerits	Factor	Rating
End Cover	2.0	1	2.0
Spacer and Nut	9.5	2	19.0
Heater - Front	21.4	3	64.2
Heater - Rear	15.2	3	45.6
Seal Plate	10.0	1	10.0
Bearing	8.5	5	42.5

OVERALL RATING: $\frac{183.3}{6} = 30.6$

TABLE 10

MIL-L-27502 BEARING TEST, MCS 1709
OIL PERFORMANCE

Hours	Viscosity (cs) @ 210°F	Viscosity (cs) @ 100°F	Viscosity Increase (%) @ 100°F	Total Acid No.
00:00	6.7	39.6	-	0.08
04:00	6.8	40.1	1.3	0.67
08:00	6.9	41.6	5.1	0.67
12:00	7.4	43.2	9.1	0.67
16:00	7.4	44.4	12.1	0.73
20:00	7.4	45.0	13.6	0.73
24:00	7.5	45.9	15.9	0.76
28:00	7.6	47.0	18.7	0.79
32:00	7.7	47.6	20.2	0.81
36:00	7.7	48.3	22.0	0.81
40:00	7.8	49.5	25.0	0.84
44:00	7.8	50.4	27.3	0.90
48:00	7.8	50.8	28.3	1.04

VISCOSITY INCREASE, 48 hr. (cs)	
@ 100°F	11.2
@ 210°F	1.1
VISCOSITY INCREASE, 48 hr. (%)	
@ 100°F	28.3
@ 210°F	16.4
TOTAL ACID NUMBER INCREASE	0.96

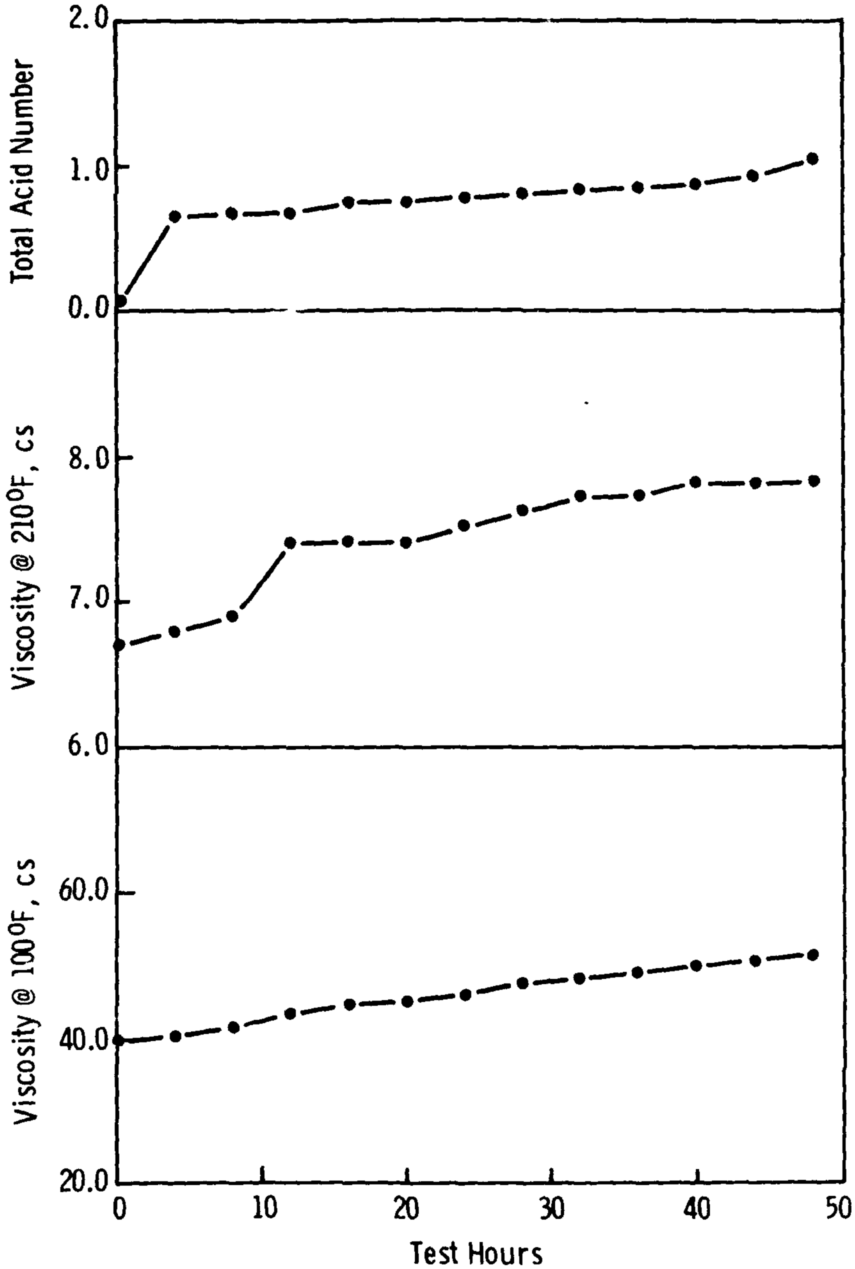

Figure 4. Plot of Viscosity and Total Acid Number vs Time for MCS 1709. (MIL-L-27502 Bearing Test)

VI. RYDER GEAR LOADS OF MCS 1710 AND MCS 1709

Ryder gear tests were run on MCS 1710 and MCS 1709 by Alcor, Inc., of San Antonio, Texas. This test (ASTM D-1947) measures the gear-load-carrying ability of a lubricant; it involves putting stepwise loads on AMS 6260 spur gears and observing the tooth face scuffing at each load increment. The load-carrying capacity of the oil is the tooth load (in pounds per inch of tooth face width) at which an average tooth face scuffing of 22.5% occurs.

The average rating for MCS 1710 and MCS 1709 was 3000 lb/in. and 3200 lb/in. respectively. The MIL-L-27502 specification for two determinations is 2550 lb/in. minimum. More details are given below:

	MCS 1709	MCS 1710
A-side rating, lb/in.	3180	3030
B-side rating, lb/in.	3220	2970
Average rating, lb/in.	3200	3000
Relative rating, % of Ref. Oil C	110.0	103.1

VII. CONTROL OF THE MAGNESIUM CORROSION OF MCS 1710

A. Metal Deactivators

Three metal deactivators tested in MCS 1710 effectively passivated magnesium at concentrations of 0.1% to 0.01% or lower. Since these additives are proprietary they are simply designated in this report as Deactivator 1, 2 and 3. At the lower concentration Deactivator 2 and 3 did not produce any sludge during 464°F oxidation-corrosion tests. Moreover, they showed promising initial storage life at both 65°C and 100°C. The data are presented in Table 11. In micro oxidation-corrosion tests run at AFML, Deactivator 1 at 0.1% inhibited magnesium corrosion for 72 hours (weight loss = 0.02 mg/cm^2). At 0.05% concentration there was no metal corrosion after 48 hours and a weight loss of -2.8 mg/cm^2 after 72 hours.

Because of its effectiveness in minimizing magnesium corrosion and because of its good storage life, it was decided to add Deactivator 3 to MCS 1710 in the 200-gal sample prepared for AFML.

B. Identification of the Corroding Additive in MCS 1710

A regression analysis of oxidation-corrosion test data showed that the corrosion of magnesium is accentuated by an interaction between two additives in MCS 1710. Eliminating one of these additives reduced the corrosion but did not eliminate it. Storage problems were probable if the second additive was removed from the blend, so no further work using this approach was done. The analysis is described in detail in Appendix C.

TABLE 11

PROPERTIES OF MCS 1710 CONTAINING METAL DEACTIVATORS

Deactivator	Conc. (wt%)	Mg Change @ 428°F (mg/cm^2)	Sludge at 464°F	Storage Life (days)** 65°C	100°C
1	0.1	+0.02	light	>111	7
	0.07	+0.05	none	42	6
	0.01	+0.14	light	> 74	>74
2	0.1	+0.05	light	>104	9
	0.05	+0.14	light	>104	9
	0.01	+0.14	none*	3	>59
	0.005	-0.10	none*	18	>63
3	0.1	+0.13	not run	> 83	14
	0.01	-0.12	none*	> 83	>83
	0.005	-0.22	not run	> 64	>64
none	-	∿-13	none		

*Test ran for 64 hours, the tubes were withdrawn from the block for ∿3 hours, then run @ 464°F for another 8 hours.

**Storage data through 7/22/75.

VIII. 200-GALLON SAMPLE OF MCS 1710A

Two hundred gallons of MCS 1710 containing metal Deactivator 3 at a concentration of 0.01% was blended and forwarded to AFML. This large batch was equivalent in oxidation-corrosion test performance to previously prepared MCS 1710A except it showed higher magnesium corrosion at 428°F (-1.0 mg/cm^2 vs. -0.12 mg/cm^2). One possible cause for this increase was contamination of the sample during the blending in plant equipment. To test this, we prepared in the laboratory a blend of MCS 1710A containing the same additives used in the 200-gal lot. This gave a magnesium corrosion (-1.4 mg/cm^2), similar to that of the 200-gal sample, indicating that base stock contaimination during the large-scale preparation was not causing the corrosion. A sample from the 200-gal blend containing 0.02% metal deactivator showed a magnesium loss of 0.7 mg/cm^2. This is still above the MIL-L-27502 specification of ±0.2 mg/cm^2. These results are summarized in Table 12. Tables B1 and B2 of Appendix B give the remaining oxidation-corrosion data on the 200-gal sample.

TABLE 12

MAGNESIUM CORROSION BY MCS 1710A

	Magnesium Change at 428°F (mg/cm^2)(72 hr)
MCS 1710	-13
MCS 1710A (lab sample)	- 0.1
MCS 1710A (200 gal)	- 1.0
MCS 1710A (additives used in 200-gal, lab blending)	- 1 4
MCS 1710A + 0.01% excess metal deactivator	- 0.7

IX. STORAGE TEST DATA

Most of the storage data in Table 13 are for various additives in Fluid 6* as of July 22, 1975. The deposit inhibitors are proprietary and are given code numbers. They are combined with a standard amine from MCS 1034 and dioctyldiphenyl amine. All of these combinations show promising storage; MCS 1710 is particularly good. There is a noticeable storage improvement in changing from Fluid 6 to Fluid 12**.

As mentioned earlier, Fluid 12 is no longer commercially available. Storage data on a replacement ester, L-100, are very promising.

*90% L-78, 10% L-31
**95% L-71, 5% L-31

TABLE 13

STORAGE TEST DATA

Deposit Inhibitor	Days to Appearance of Solids in the Fluid		
	65°C	100°C	150°C
Standard	351	27	
8 (double conc.)	>652	367	
9	585 >538	>680 70	
9 (double conc.)	652	415	
27	160 >379	>490 >379	
39	>445 63 >379	441 10 >379	
MCS 1709	>652 >363 7 16 192	550 >363 >376 63 >217	
MCS 1710	>405 >378 >356 >291 >201	>405 >378 287 118 >265	
MCS 1710A	>83 >32	>83 >32	
Standard (in Fluid 12)	>760	>760	
Standard (in L-100)	>151 >109 >107	>151 >109 >107	>151 >109 >107
Additives of MCS 1710 minus triphenylphosphine oxide (in L-100)	>151 >107	>151 >107	120 >107

APPENDIX A

COMPARISON OF FEDERAL 5307 OXIDATION-CORROSION TEST AND THE MODIFIED 5307 TEST

The oxidation-corrosion tests used in this contract were run according to Federal Test Method Standard 791b, Method 5307, with only two changes:

1) The post-test metal specimens were cleaned by soaking in acetone and scrubbing with a stiff bristle brush to remove corrosion and deposits. This method is preferred over electrocleaning.

2) The descriptions of deposit formations were from direct visual observation only. No centrifugal analyses of solids were run. Coke is the solid matter above the fluid sample; sludge is the solid material formed in the fluid.

The tests used in the initial contract had further modifications which are described in the final report (Ref. 1). The differences between the test techniques are as follows:

5307	Modified 5307
Measures fluid temp. (~10°F lower than the block temp.)	Measures block temp.
200-g samples (six 10 ml aliquots withdrawn during the test)	125-g samples
Water condenser cooling	Air condenser cooling (72°F)
Round coupons (~0.39 in.^{2})	Square coupons (1 in.^{2})
51-mm diameter tubes	70-mm diameter tubes
6-mm inlet tube	7-mm inlet tube tapered to 2-mm at the end

Block vs. Fluid Temperature

The fluid temperature in the oxidation tests was consistently around 10°F lower than the block temperature.

Block Temperature, °F	Fluid Temperature, °F
464	455 ± 1
428	418

This ten degree range is in good agreement with values in the literature (Ref. 2, pg. 22). It is ascribed to the warming of the cold gas by the oil and limited heat transfer from the block to the oil. To run a test at a given temperature, the oil must be 10° hotter in the 5307 test than in the modified procedure. This will obviously accelerate oxidative reactions. The literature (Ref. 2, pg. 9) estimates that an 18°F temperature rise doubles the rate of a high temperature oxidation.

Sample Size, Tube Size

The data below (from the first contract) show that increasing the sample size makes the modified procedure less severe.

TABLE A1

OXIDATION-CORROSION TESTS ON
DIFFERENT SAMPLE SIZES OF FLUID 12*
455°F Fluid Temperature, 72 hr, 10 liters air/hour

Sample Size	Visc. Inc. (%)	Acid No.	Bronze II Change (mg/cm^2)
125 g	105.2	12.8	-0.4
250 ml	35.9	6.3	-0.2

Presumably, then, the larger sample size of the 5307 test makes that test less severe compared with the modified test. This is surprising in that a larger sample will increase the time of contact between the air bubbles and the fluid and thus increase the percent of oxygen absorbed (Ref. 2, pg. 14). Likewise, using a narrower tube (5307 method) should increase the time of contact.

*Containing the standard additives

Condenser Cooling

The federal 5307 method is not precise about the condenser temperature, specifying only that the water cooling be below 81°F (27°C). In this contract, some tests were run during the winter and the condenser water temperature was $\sim$37°F. Finally, to standardize this variable, the water was held at 59-68°F (15-20°C) during later runs. The oxidation tests of the first contract used cooling ($\sim$72°F).

The temperature of the vapor space above the oil will influence the oxidation by changing the amount of vapor phase oxidation and by controlling the amount of volatiles returned to the oil. A comparison between a heated jacket and an air cooled condenser documents these effects (Ref. 2, pg. 23). The vapor temperature ranges covered in the above procedures would not produce much vapor oxidation. However, changes in the return of volatiles may have altered some of the test results.

Inlet Tube

The narrow inlet used in the modified procedure should produce smaller bubbles at a constant air flow and a greater area of air-oil interface.

The two procedures do not always give similar results; there is no single direction in the variables between them, and a simple correlation is not feasible. Generally, the 5307 method gave more severe metal corrosion but smaller viscosity increases than the modified procedure.

APPENDIX B

DETAILED RESULTS OF 5307 METHOD OXIDATION-CORROSION TESTS ON CANDIDATE OILS: COMPARISON WITH DATA FROM OUTSIDE LABORATORIES

All tests used two tubes of each fluid. The reported values in Tables B1 and B2 are averages of the individual tubes from a typical run.

Forty-eight-hour 5307 oxidation-corrosion tests on MCS 1710 were run for AFML. A comparison with samples from one of the MRC runs is given in Table B3; the agreement is quite good. Table B4 compares oxidation-corrosion tests (5307 method) on MCS 1710A (2 gal sample) run at MRC and at Alcor, Inc. The 428°F tests were run concurrently; the MRC data at 464°F are from Table B3.

The reproducibility is very good except for the high magnesium corrosion in the 48-hour Alcor test. This did not occur in the 72-hour Alcor test or in either MRC test. No significant bronze or steel corrosion was observed in any test.

TABLE B1

428°F OXIDATION-CORROSION TESTS (72 HOURS)

	Visc. Inc. (%)	Acid No.	Metal Wt. Change (mg/cm^2) Al	Ag	BrI	Fe	M-50	Mg	Ti	Deposits
MCS 1710	18.4	4.4	+0.0	0.0	+0.0	-0.2	+0.0	-14.2	-0.0	V.lt. coke No sludge
MCS 1710A (lab sample)	13.5	0.7	+0.0	-0.0	-0.0	+0.0	+0.0	- 0.1	+0.0	Lt. coke No sludge
MCS 1710A (200 gal)	12.5	0.3	-0.0	-0.1	-0.1	-0.0	-0.0	- 1.0	-0.0	V.lt. coke No sludge
MCS 1709	11.4	1.5	-0.0	-0.0	+0.0	+0.0	NC	- 0.2	+0.0	V.lt. coke No sludge
Fluid 12	9.4*	1.4	+0.0	0.0	+0.0	-0.1	+0.0	- 0.1	-0.0	V.lt. coke No sludge

*Wide variations (see Table 1)

TABLE B2

464°F OXIDATION-CORROSION TESTS (72 HOURS)

	Visc. Inc. (%)	Acid No.	Metal Wt. Change (mg/cm^2) Al	Ag	BrII	Fe	M-50	WSP	Ti	Deposits
MCS 1710	29.1	6.3	-0.0	+0.1	+0.0	-0.0	-0.0	-0.0	+0.0	Lt. coke No sludge
MCS 1710A (lab sample)	28.5	5.7	-0.1	-0.1	+0.1	+0.1	+0.1	+0.1	+0.1	Lt. coke No sludge
MCS 1710A (200 gal)	24.0	5.7	0.0	-0.0	-0.0	0.0	0.0	-0.0	+0.0	V.lt. coke No sludge
MCS 1709	43.4	7.8	-0.0	+0.1	-0.2	+0.1	+0.1	+0.0	-0.0	Lt. coke No sludge
Fluid 12	63.5*	12.3	-0.0	-0.1	-0.1	+0.1	+0.1	+0.1	+0.1	Lt. coke No sludge

*Wide variations (see Table 2)

TABLE B3

COMPARISON OF MONSANTO AND AFML OXIDATION-CORROSION DATA ON MCS 1710 (5307 METHOD) (48 HOURS)

	AFML	Monsanto
464°F		
Viscosity Increase (%)	19.7	20.4
TAN	2.4	1.8
428°F		
Viscosity Increase (%)	10.2	8.7
TAN	0.2	0.3

TABLE B4

COMPARISON OF MRC AND ALCOR 5307
OXIDATION-CORROSION TESTS ON MCS 1710A

	Alcor	MRC
Corrosion-Oxidation Stability @ 428°F		
72 hr		
Corrosion (mg/cm^2):		
Steel	+ 0.0	+ 0.0
Silver	- 0.1	- 0.1
Aluminum	NC	- 0.0
Magnesium	- 0.6	- 0.4
Bronze, AMS 4616	+ 0.0	- 0.1
Titanium	- 0.0	+ 0.0
M-50 Steel	- 0.0	- 0.0
Oxidation:		
Viscosity Change @ 100°F, (%)	14.5	11.2
Total Acid Number, Final	0.2	0.4
48 hr		
Corrosion (mg/cm^2):		
Steel	NC	NC
Silver	- 0.0	- 0.0
Aluminum	- 0.0	NC
Magnesium	-11.6	- 0.3
Bronze, AMS 4616	- 0.0	- 0.1
Titanium	- 0.0	+ 0.0
M-50 Steel	- 0.0	- 0.0
Oxidation:		
Viscosity Change @ 100°F, (%)	10.9	7.6
Total Acid Number, Final	1.5	0.4
Corrosion-Oxidation Stability @ 464°F		
72 hr		
Corrosion (mg/cm^2):		
Steel	- 0.0	NC
Silver	NC	- 0.0
Aluminum	+ 0.0	NC
Waspaloy	NC	- 0.0
Bronze II	- 0.1	- 0.0
Titanium	NC	+ 0.0
M-50 Steel	+ 0.0	NC
Oxidation:		
Viscosity Change @ 100°F, (%)	26.0	24.0
Total Acid Number, Final	5.4	5.7
48 hr		
Corrosion (mg/cm^2):		
Steel	+ 0.0	NOT RUN
Silver	- 0.0	
Aluminum	+ 0.1	
Waspaloy	- 0.0	
Bronze II	- 0.0	
Titanium	+ 0.0	
M-50 Steel	- 0.1	
Oxidation:		
Viscosity Change @ 100°F, (%)	17.8	20.4
Total Acid Number, Final	1.5	1.8

APPENDIX C

IDENTIFICATION OF THE CORRODING ADDITIVES IN MCS 1710 BY REGRESSION ANALYSIS

Three of the six additives present in MCS 1710 are also in other ester blends which do not corrode magnesium. Assuming no additive interactions, the corrosion of MCS 1710 is caused by the moieties in MCS 1710 which are not in the other fluids. Calling the "non-corroding" additives, a_1, b_1 and c_1 and the "corroding" additives a_2, b_2 and c_2, the grid below represents all the possible additive combinations.

	a_1		a_2	
	b_1	b_2	b_1	b_2
c_1	x			x
c_2		x	x	

Running the x-marked squares should identify the corroding additive. The corrosions of the base stock (Fluid 6) containing the following additives were measured:

a_1	b_1	c_1	(blend 1)
a_1	b_2	c_2	(blend 2)
a_2	b_1	c_2	(blend 3)
a_2	b_2	c_1	(blend 4)

(If a_2 were the corroding additive, then blends 3 and 4 would corrode but 1 and 2 would not, etc.)

The results, given in Table C1, show that <u>all of the blends corroded magnesium</u>. This suggested additive interactions. Regression analysis of these data plus data from other runs on these additives pinpointed this interaction. All of the combinations corrode magnesium, but the corrosion is accentuated by an interaction between a_2 and c_2.

The equation from the analysis is:

$$\text{Mg corrosion} = 4.32 + 7.5 \text{ (if } a_2 \text{ and } c_2 \text{ are } \underline{\text{both}} \text{ present)}$$
$$(\sigma = 2.3 \text{ at } 99.9\% \text{ confidence})$$

That is, Mg corrosion is 11.82 mg/cm^2 if a_2 and c_2 are both present, and 4.32 mg/cm^2 if they are not.

The data used are given in Table C2.

We tried to eliminate the corrosion of MCS 1710 by dropping c_2 from the additive package. This reduced the corrosion from -14.4 mg/cm^2 to -6.7 mg/cm^2 but did not totally stop it. Since dropping a_2 will probably lead to storage problems, no further work with this approach was attempted.

TABLE C1

MAGNESIUM CORROSION OF MODEL BLENDS

Blend	Mg Loss (mg/cm^2)	
1	5.9	average values from two tubes
2	5.4	
3	11.2	
4	4.2	

TABLE C2

MAGNESIUM CORROSION DATA FOR REGRESSION ANALYSIS

Additives (in Fluid 6)			Mg Loss (mg/cm^2) (per tube)
a_1	b_1	c_1	5.0, 6.8
a_1	b_1	c_1	6.4, 5.5
a_1	b_1	c_1	0.1, 0.3
a_1	b_2	c_2	5.2, 5.4
a_2	b_1	c_2	10.7, 11.3 (11.7)
a_2	b_2	c_1	5.8, 2.6
a_2	b_2	c_2	8.3 (8.7), 11.9
a_2	b_2	c_2	13.7, 14.8

REFERENCES

1. Clark, F. S., Miller, D. R. and Reid, S. L., "Development of a Gas Turbine Engine Oil for Bulk Oil Temperatures of -40 to 464°F," AFML-TR-74-247, Final Report, Contract No. F33615-73-C-5079, Monsanto Research Corp. Report No. MRC-SL-477, April 1975.

2. Klaus, E. E., Tewksbury, E. J., et al, "Fluids, Lubricants, Fuels and Related Materials," AFML-TR-74-201, Part I, June 1974.